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## CLAIMS

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### [Claim(s)]

[Claim 1] The sulfonation approach characterized by recognizing specified quantity existence of the alicyclic unsaturated hydrocarbon in case the resin containing a styrene system polymer is sulfonated in a solvent.

[Claim 2] the above-mentioned styrene system polymer -- a styrene unit -- a unit -- more than total 30 mol % -- the sulfonation approach according to claim 1 characterized by containing and the whole resin containing 20% of the weight or more.

[Claim 3] The sulfonation approach according to claim 1 characterized by the resin containing the above-mentioned styrene system polymer being used scrap wood.

[Claim 4] The sulfonation approach according to claim 1 that the above-mentioned alicyclic unsaturated hydrocarbon is characterized by being alicyclic six membered ring-like unsaturated hydrocarbon.

[Claim 5] The sulfonation approach according to claim 1 characterized by making the 0.01 - 5 % of the weight of the above-mentioned alicyclic unsaturated hydrocarbon exist to a styrene system polymer.

[Claim 6] The sulfonation approach according to claim 1 characterized by being that in which the resin containing the above-mentioned styrene system polymer contains alicyclic unsaturated hydrocarbon beforehand.

[Claim 7] The sulfonation approach according to claim 6 that the resin containing the above-mentioned styrene system polymer is characterized by contracting and collecting styrene foam with alicyclic unsaturated hydrocarbon.

[Claim 8] The sulfonation approach according to claim 7 characterized by the above-mentioned alicyclic unsaturated hydrocarbon being a limonene.

[Claim 9] The sulfonation approach according to claim 1 characterized by the resin containing the above-mentioned styrene system polymer containing an inorganic pigment.

[Claim 10] The sulfonation approach according to claim 8 that the above-mentioned inorganic pigment is characterized by being carbon black and/or titanium oxide.

[Claim 11] The sulfonation approach according to claim 8 characterized by the above-mentioned inorganic pigment containing 0.01 to 10% of the weight to the resin containing a styrene system polymer.

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134

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the sulfonation approach of a polystyrene system polymer.

[0002]

[Description of the Prior Art] Polystyrene is cheap, lightweight, and since it excels in properties, such as electrical characteristics, and rigidity, a water resisting property, it is used abundantly as resin ingredients, such as a packing material, shock absorbing material (styrene foam), electric appliances, and a box of an automobile, various components ingredients.

[0003] Therefore, together with the polyolefine system resin represented by polyethylene, it is produced so much as versatility resin.

[0004] In such a situation, the further application amplification of polystyrene system resin is expected, and conversion in an object with more high added value is considered.

[0005] On the other hand, the place which the yield of the scrap wood as dust after an activity also increases every year comes with the increment in the volume of polystyrene system resin, and 2-ZU about a deployment of these scrap wood has been increasing from the rise of the interest of earth environmental protection in recent years.

[0006]

[Problem(s) to be Solved by the Invention] The technique which embellishes polystyrene system resin chemically and is reformed on the polymer coagulant for waste water treatment, the admixture for cement, the additive for papers, the dispersant for magnetic powder, ion exchange resin, etc. as an approach of using polystyrene system resin for the high application of added value other than a structural material is proposed.

[0007] This technique sulfonates polystyrene system resin in a solvent, and reforms to a polyelectrolyte.

[0008] However, in this system of reaction, since the sulfonated polystyrene which is a product deposits by the shape of a slurry in a solvent during a reaction, the phenomenon in which a mechanical load is applied to an agitator with progress of a reaction has arisen.

[0009] From such a situation, a technique which can make reaction concentration high for improvement in productive efficiency is desired.

[0010] This invention is proposed in view of such the actual condition, and aims at improving the sulfonation technique of polystyrene system resin. It aims at offering the sulfonation approach which it can sulfonate by high concentration and can specifically reform polystyrene system resin (scrap wood) to a polyelectrolyte at stability.

[0011]

[Means for Solving the Problem] As a result of repeating research wholeheartedly with what does not conquer the above-mentioned technical problem, when this invention person sulfonates polystyrene system resin in a solvent, he is adding alicyclic unsaturated hydrocarbon to the system of reaction, finds out that the sulfonation reaction in high concentration is attained, and came to complete this invention.

[0027] If it applies to the scrap wood which contracted and collected styrene foam with alicyclic

134

unsaturated hydrocarbon, for example, a limonene, especially, it is not necessary to newly add alicyclic unsaturated hydrocarbon in the case of sulfonation, and the consistent system can realize from recovery of styrene foam to reuse-ization.

[0028] Since this can be used also in a sulfonation reaction as a contraction agent of styrene foam as it is when a limonene is used although isoamyl acetate besides a limonene, benzyl propionate, ethyl butylate, etc. are usable, it is advantageous. It is the liquid constituent which added the ethanol of 1 - 6 volume % to the limonene most preferably.

[0029] As the refining approach of polystyrene system resin, a sulfonic group or a sulfonium salt radical can be used as a polyelectrolyte by introducing as a substituent at the above-mentioned polystyrene system resin.

[0030] What is necessary is just to add water and a basic water solution, respectively, after adding a sulfonation agent to what dissolves or distributed polystyrene system resin in the organic solvent and performing a sulfonation reaction to it as an approach of introducing a sulfonic group or a sulfonium salt radical.

[0031] As a sulfonation agent, a sulfuric anhydride, an oleum, a chlorosulfonic acid, and concentrated sulfuric acid are mentioned. These sulfonation agent may be used independently, respectively and may be used together. [ two or more ]

[0032] Moreover, it is the 50-100-mol range of % suitably [ using 40-200 mol % to the aromatic series unit containing the styrene in polystyrene system resin as the addition ], and still more preferably.

[0033] If there are few additions than this, whenever [ sulfonation ] will become low and the property as a polyelectrolyte will no longer be acquired. On the contrary, when many [ too ], it becomes easy to produce sulfone bridge formation, and a reaction becomes an ununiformity or by-products, such as a sulfate, will remain so much in the system of reaction.

[0034] The above-mentioned sulfonation agent may use together with a Lewis base. As a Lewis base, alkyl phosphate (triethyl phosphate, trimethyl phosphate), dioxane, an acetic anhydride, a \*\*\*\*\* chill, PAL thymine acid ethyl, diethylether, thioxan, etc. are mentioned.

[0035] the addition pile of these Lewis bases -- the styrene unit in polystyrene system resin -- receiving - 1-200-mol % -- it is 2-100-mol % preferably. In addition, if there are few additions of a Lewis base, it will become easy to generate a gelation object during a sulfonation reaction, and if many, yield will fall in \*\* that the sulfonation reaction itself cannot advance easily.

[0036] As a reaction solvent used for the above-mentioned sulfonation reaction time, they are the aliphatic series halogenated hydrocarbon (preferably 1,2-dichloroethane, chloroform, dichloromethane, 1,1-dichloroethane) of carbon numbers 1-2, aliphatic cyclic hydrocarbon (preferably a cyclohexane, a methylcyclohexane, a cyclopentane), nitromethane, 2 TOROPENZEN, and sulfur dioxide.

[0037] In addition, these solvents may be used with a the very thing simple substance, and more than one may be mixed and they may be used. In mixing within the above-mentioned solvent, especially a limit does not have the mixed ratio.

[0038] Or more than one may be mixed with other solvents. As a solvent which can be mixed and used, paraffin hydrocarbon (carbon numbers 1-7), an acetonitrile, a carbon disulfide, a tetrahydrofuran, tetrahydropyran, 1, 2-dimethoxyethane, an acetone, a methyl ethyl ketone, a thiophene, etc. are mentioned. They are paraffin hydrocarbon (carbon numbers 1-7), a tetrahydrofuran, an acetone, and an acetonitrile preferably in these things. Especially the mixed ratio with a solvent besides these is not limited.

[0039] Moreover, after reaction termination, the solvents once used for the above-mentioned reaction may be collected by approaches, such as sampling and distillation, and may be again used for a reaction.

[0040] 0-100 degrees C of temperature of the above-mentioned sulfonation reaction are 15-80 degrees C preferably. If temperature is lower than this range, a sulfonation reaction will stop being able to go on easily and yield will fall.

[0041] The time amount (however, the drop time of a sulfonation agent is not included) of a sulfonation reaction is 30 minutes - 5 hours preferably for 10 minutes to 10 hours.

[0042] The polymer concentration in the sulfonation system of reaction is 0.1 - 50 % of the weight, and is 5 - 40 % of the weight preferably. If concentration is lower than this range, productive efficiency and the rate of installation of the sulfone radical to a polymer will fall. Moreover, if concentration is high, churning of the system of reaction will become difficult, or it becomes easy to generate the side reaction of sulfone bridge formation.

[0043] This invention makes it possible to make homogeneity distribute the slurry which is the product which deposits in the system of reaction at the time of the above-mentioned sulfonation. Therefore, reaction concentration can be raised. The sulfonation object of the alicyclic unsaturated hydrocarbon used as the dispersant of a product slurry is made to generate in this invention by making alicyclic unsaturated hydrocarbon exist in the sulfonation system of reaction.

[0044] As the above-mentioned alicyclic unsaturated hydrocarbon, a six membered ring-like thing is desirable and monocycle type monoterpenes (a limonene, terpinene, etc.), 2 ring type monoterpenes (Caren, a pinene, sabinene, camphene, etc.), terpinolene, a cyclohexene, a monoalkyl (carbon numbers 1-4) cyclohexene, a terpeneol, etc. are specifically mentioned.

[0045] In these, they are a monocycle type monoterpene, a cyclohexene, and a methyl cyclohexene preferably (a limonene, sylvestrene, terpinene, etc.).

[0046] The addition to the sulfonation system of reaction of the above-mentioned alicyclic unsaturated hydrocarbon is 0.05 - 1.0 % of the weight preferably 0.01 to 5% of the weight to a styrene system polymer.

[0047] If there are few additions of alicyclic unsaturated hydrocarbon than this, the dispersion effect of a slurry will be lost. Moreover, in many [ conversely / too ], many alicyclic unsaturated hydrocarbon sulfonation objects of a by-product will be generated, and become disadvantageous in respect of quality and cost (lowering of purity).

[0048] In addition, although it is desirable to add to sulfonation reaction before or reaction time as for alicyclic unsaturated hydrocarbon, it may be beforehand sulfonated in polystyrene system resin, using as a raw material what alicyclic unsaturated hydrocarbon contains. For example, when the resin recycled article collected from the styrene foam contracted by the limonene as a raw material is used, alicyclic unsaturated hydrocarbon will be contained in a raw material.

[0049] This invention is making alicyclic unsaturated hydrocarbon exist in sulfonation reaction time in a system, and this compound is sulfonated and it aims at improvement in the dispersibility of a slurry by working as a surfactant to the slurry this thing of whose is a product. This becomes possible to raise the reaction concentration of sulfonation reaction time.

[0050] In addition, if inorganic pigments, such as carbon black and titanium oxide, exist in the system of reaction at the above-mentioned sulfonation reaction time, the place whose dispersibility of a slurry improves further will come. Moreover, the inorganic pigment may be beforehand contained in resin as a coloring agent, and may newly be added in the system of reaction or a reactant. However, since it is easy to carry out homogeneity distribution of the direction which sulfonated what is beforehand contained in resin in an additive, it is desirable.

[0051] In addition, the content in the system of reaction of the above-mentioned inorganic pigment is 0.01 - 10 % of the weight to a resinous principle. If the dispersion effect of a slurry is conversely high low when there are few contents, sulfonation conversion will fall.